Mechanical and Thermal Properties of Talc and Calcium Carbonate Filled Polypropylene Hybrid Composites

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ABSTRACT: The main aim of this work was to study and compare the mechanical and thermal properties of hybrid polypropylene (PP) composites and single-filler PP composites. With two main types of mineral fillers—calcium carbonate (CaCO₃) and talc—PP composites of different filler weight ratios (talc/CaCO₃) were compounded with a twinscrew extruder and then injection-molded into dumbbell specimens with an injection-molding machine. Tensile, flexural, and impact tests were performed to determine and compare the mechanical properties of the hybrid and single-filler PP composites. A synergistic hybridization effect was successfully achieved; the flexural strength and impact

strength were highest among the hybrids when the PP/talc/ CaCO₃ weight ratio was 70:15:15. The nucleating ability of the fillers and its effects on the mechanical properties were also studied with differential scanning calorimetry. Because of the influence of talc as the main nucleating agent, the hybrid fillers showed significant improvements in terms of the nucleating ability, and this contributed to the increase in or retention of the mechanical properties of the hybrid composites. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 91: 3327–3336, 2004

Key words: poly(propylene) (PP); composites; fillers

INTRODUCTION

Polypropylene (PP) has been known for its good mechanical properties and processability, which allow it to accept numerous types of natural and synthetic fillers. Its versatility has also led to the possibility of producing particulate-filled composites.

The incorporation of fillers such as mica, kaolin, calcium carbonate (CaCO₃), and talc into thermoplastics is a common practice in the plastics industry, used to reduce the production costs of molded products. Fillers are also used to improve the working properties of thermoplastics, such as the strength, rigidity, durability, and hardness.¹ High filler loadings, however, may adversely affect the processability, ductility, and strength of composites. The incorporation of additives such as elastomers, coupling agents, compatibilizers, surface agents, antioxidants, and ultraviolet agents has also been performed to further enhance the durability and appearance of composites.²

Works on single-filler PP composites are abundant and can be found in almost every polymer and composite journal. Typical fillers and reinforcements that are incorporated into PP matrices include glass fibers,^{3,4} natural fibers and fillers,^{5–7} and mineral fillers such as talc and $CaCO_3$.^{1,8–17} The effects of these individual fillers on the properties of PP-based components are relatively well known. As for fillers, investigations of CaCO₃ and talc have been very thorough because of their widespread use in industry. The introduction of talc has been known to improve some properties of PP, such as the strength, dimensional stability, stiffness, and crystallinity, but it has a detrimental effect on other properties, such as the impact strength and deformability. CaCO₃, however, imparts high impact properties and deformability at the cost of the strength. Although the facts and figures of both talc and CaCO₃ are well established, these works have only focused on the characterization of single-filler systems, and not much has been done to introduce a combination of these fillers into one matrix.

Previously, reinforced ternary polymer blends have been reported.^{18–22} There have also been publications on hybrid composites with two different kinds of reinforcements in the form of natural fibers, glass fibers, carbon fibers, talc, mica, and glass beads.^{23–26} However, the effects of incorporating two types of particulate fillers simultaneously into the PP matrix have not been, to our knowledge, systematically studied. Therefore, it is obvious that incorporating a mix of both fillers with different compositions into the PP matrix, which yields hybrid composites, can improve the properties of PP through the combination of the advantageous properties of the two fillers. These hybrids can achieve synergistic hybridization effects,

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with an improvement of mechanical properties, at a reduced cost. Hybrid composites are becoming more and more commercially significant in their own right for a number of reasons. First, there are economic advantages in diluting a more expensive reinforcement or filler with cheaper materials. Second, a wider spectrum of physical and mechanical properties is possible, facilitating the design of materials with specific characteristics. Third, hybrids are used to achieve synergistic effects and improvement in mechanical characteristics and functional properties.

Of particular interest to us are the effects of the incorporation of different mineral fillers (CaCO₃ and talc) into the PP matrix on the mechanical properties of the composite. Our aim is to ultimately replace at least 50% of talc with $CaCO_3$, because the latter is much cheaper than talc, while the mechanical properties of the hybrids are maintained. A preliminary study was done on PP composites filled with different contents of talc and CaCO₃.¹⁷ Increased rigidity of the materials was observed as a function of both filler amounts, whereas the highest mechanical strength was obtained at a particular content of talc. Here we report the preparation of PP composites with combined fillers of talc and CaCO₃. The particle dispersion and changes in the microstructure have also been studied to determine the factors governing the mechanical behavior of the composites. Talc is known to impart stiffness and strength to composites. However, this is usually attained at the expense of impact strength. According to Katz and Milewski,²⁷ CaCO₃filled PP appears to have higher impact strength, brighter color, higher thermal stability, improved fatigue strength, and lower stiffness in comparison with a talc-filled grade. In this respect, the introduction of CaCO₃ into talc-filled PP, in addition to making the composite cheaper, has the unique advantage of synergistically increasing the overall impact strength of the hybrid composite without compromising the other mechanical properties much. CaCO₃ treated with stearic acid was also used in this study.

EXPERIMENTAL

Materials

Copolymer-grade PP (Pro-Fax SM240), with a melt index of 25 g/10 min and a density of 0.894 g/cm³, was supplied by Titan PP Polymers (M) Sdn. Bhd.

The fillers were talc (Chung Chemicals Sdn. Bhd.) and untreated (Omyacarb 3-SA) and stearic acid treated (Omyacarb 3-TSA) CaCO₃ (Malaysian Calcium Corp. Sdn. Bhd.). Table I lists the specifications.

The additives included an antioxidant (Irganox 1010) and an ultraviolet stabilizer (Tinuvin 770 DF), both from Ciba.

TABLE I Material Specifications

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Material	Density (g/cm ³)	Hardness (Moh's scale)	Mean particle diameter (µm)
Talc	2.79	1	6.3
Untreated CaCO ₃	2.70	3	4.5
Treated CaCO ₃	2.70	3	4.5

Sample preparation

The compounding was performed with a Rheomex CTW 100 twin-screw extruder (Haake). The barrel temperatures of the four zones were 160, 170, 180, and 190°C, from the feeding zone to the die zone. The compounds were blended at a screw speed of 25 rpm. The extrudates were cooled in a water bath, pulled, and pelletized. Test specimens were injection-molded in a tensile mold with a Battenfeld BA 350 CD 35-ton machine with a Unilog 4000 control system. The injection-molding temperature ranged from 190 to 230°C, and the back-pressure was 60 psi.

Mechanical testing

The tensile and flexural properties were measured with a Testometric M500-25 kN tensile machine in accordance with ASTM D 638 and ASTM D 790-86 with type 1 test specimen dimensions. For tensile tests, a crosshead speed of 5 mm/min was used, and the average value of at least five specimens was taken for each sample. For flexural tests, a three-point loading system was used, and the support span length was adjusted to 50 mm. The crosshead speed was 3 mm/ min. All tests were carried out in an air-conditioned room (27° C).

Impact testing was performed with a Ray-Ran impact pendulum tester at an impact energy of 7.5 kJ according to ASTM D 256-88. The hammer velocity was set at 2.88 m/s, and the weight of the hammer was 1.811 kg.

Mode of failure

The mode of fracture was studied by an analysis of the fracture surfaces of tensile samples. This was carried out with a scanning electron microscopy (SEM) machine (Cambridge S-360, Leica); the fracture surfaces of the tensile test specimens were coated with a thin gold–palladium layer to prevent electrical charge accumulation during the examination.

Density determination

The density (ρ) of the test specimens was determined according to the ASTM D 792 water displacement method (method A) with the following equation:



Figure 1 MFI for single-filler and hybrid composites.

$$\rho = [W_1 / (W_1 - W_2)]\rho_w \tag{1}$$

where W_1 and W_2 are the sample weights in air and water, respectively, and ρ_w is the density of water.

Thermal analysis process

The crystallization behavior and melting characteristics of the composites were studied by differential scanning calorimetry (DSC) with a PerkinElmer DSC-7 in a nitrogen atmosphere at a heating and cooling rate of 20° C/min. In the first heating and cooling scans, the samples were heated from 50 to 220° C and held at that temperature for 1 min to eliminate the thermal history; then, the nonisothermal crystallization process was recorded from 220 to 50° C, and a standard status of crystallization was created.

Melt-flow index (MFI)

The processability of the filled PP composites was determined with a Ray-Ran melt-flow indexer according to ASTM D 1238-90b. A load of 2.16 kg at 230°C was used in the measurement.

Filler content determination

As some filler might be loss during processing, a polymer burn-off test or ashing was performed to determine the final filler content in the composites after the injection-molding process was completed. Dumbbells were randomly selected and burnt off in a furnace with the temperature set at 600°C. The filler residue was then weighed, and the corresponding weight fraction of the filler (W_f) was converted into the

volume fraction of the filler (V_f) with the following formula:

$$W_f = \frac{\rho_f}{\rho_c} V_f \tag{2}$$

where ρ_c and ρ_f are the densities of the composite and the filler, respectively.

RESULT AND DISCUSSION

MFI

The MFI values of single-filler and hybrid-filler composites are shown in Figure 1. Because the incorporation of fillers hinders plastic flow and increases the viscosity of a polymer melt, a decrease in MFI with the incorporation of a filler is expected. It has been noted in previous studies¹⁷ that the incorporation of CaCO₃ can retain the MFI value of a PP composite, unlike other fillers that normally reduce the MFI value of PP substantially. This signifies the ability of CaCO₃ to increase the plasticity and processability of the polymer. However, the actual mechanism leading to this phenomenon is still unclear. Although talc has the ability to slide against itself during the application of shear forces because of its platy shape, which allows an increase in the plastic flow, this ability only applies to fillers at certain parts of the composites that are near the die walls, and so the effect of this ability on MFI is minimal. Therefore, the overall MFI value of a talcfilled composite is substantially lower than the rest.

Thermal analysis

DSC analysis is one of the most convenient and accurate techniques for determining the crystallinity of a



Figure 2 Physical characteristics of the crystallization peak.

polymer composite.^{12,28} A nonisothermal crystallization approach, with a cooling rate of 20°C/min, was used to study the crystallization kinetics of the composites. A typical DSC cooling curve is shown in Figure 2.

DSC curves in Figure 3 show the effect of adding single and hybrid fillers to the crystallization kinetics of the composites. The addition of fillers clearly shows a significant increase in the temperature of the onset of crystallization (T_{o}) , which is directly correlated to the crystallinity of the composite.⁷ Talc appears to be the most efficient filler in increasing $T_{o'}$ and this confirms the strong nucleating ability of talc claimed by various researchers.^{8,12,13,16} CaCO₃, in both treated and untreated forms, also shows quite exceptional nucleating effects on PP, contrary to initial presumptions that CaCO₃ is a weak nucleating agent and a nonactive filler.⁸ The change in T_{0} of CaCO₃-filled PP composites is evident, although it is quite small. This indicates that the magnitude of its nucleating ability is small in comparison with the nucleating ability of talc. The slope of the curve for a PP composite filled with treated $CaCO_3$ is steeper than that of a composite with untreated $CaCO_3$. The increase in the nucleation rate can be attributed to improved particle dispersion caused by the treatment of the $CaCO_3$ fillers with stearic acid, which increased the number of nucleation sites and induced the nucleation rates.¹⁵

It may be inferred that the incorporation of both talc and $CaCO_3$ results in an earlier initiation of the crystallization process, which, in due course, increases the crystallinity of the PP matrix. An increase in the crystallinity brings about an increases in the modulus and strength but decreases in the elongation at break,²⁹ as shown in earlier sections of this study.

Effect of CaCO₃ and talc on the mechanical properties of PP

The various composite formulations that were used in the study are shown in Table II. To optimize the



Figure 3 Crystallization peaks for single-filler and hybrid composites.

Formulations of Single-Filler and Hybrid-Filler Composites Composition (wt %)			
PP	100:0:0		
UCC30	70:0:30		
T5UCC25	70:5:25		
T10UCC20	70:10:20		
T15UCC15	70:15:15		
T20UCC10	70:20:10		
T25UCC5	70:25:5		
T30	70:30:0		
TCC30		70:0:30	
T5TCC25		70:5:25	
T10TCC20		70:10:20	
T15TCC15		70:15:15	
T20TCC10		70:20:10	
T25TCC5		70:25:5	

 $T = talc; UCC = untreated CaCO_3; TCC = treated CaCO_3.$

appropriate ratio of the fillers, we first investigated the mechanical properties of the composites filled with a single filler of talc. The results of a previous study¹⁷ showed that with an increase in the talc content, the tensile modulus increased, the elongation at break decreased, and the tensile strength exhibited a maximum value when the concentration of talc was approximately 30 wt %. The increase in the strength of the composite at lower filler loadings could be attributed to the homogeneously dispersed talc particles because the well-dispersed particles made the crack propagation path longer, absorbed a portion of the energy, and enhanced plastic deformation. Therefore, the surface fracture energy increased as a function of the filler loading, which increased the strength of the

composite as well. However, with an increase in the talc content, the detachment of talc particles from PP caused voids, which increased in size to form a crack. In addition, the agglomeration of the filler particles resulted in a decrease in the mechanical strength because of the low strength of the agglomerates themselves.

Because the composites containing 30 wt % talc exhibited the highest tensile strength in our examination, the total filler content afterward was kept constant at 30 wt %, and CaCO₃ was used to gradually replace talc (cf. Table II).

As expected, the tensile modulus of the composites decreased (Fig. 4) with an increasing replacement of talc by $CaCO_3$ because of the lower modulus of the latter. However, the overall moduli of the composites were higher than those of unfilled PP for many reasons, such as the rigidity of the fillers and the filler particle orientation, which have been discussed in previous studies.¹⁷

The tensile strength also gradually decreased with increasing CaCO₃ content (see Fig. 5). This could be attributed to the formation of cavities around CaCO₃ particles, which could have detrimental effects on the composite strength.^{10,11} Talc, however, is a strong reinforcing filler because of its platy nature and possesses the ability to orient to the polymer flow during processing. Platy fillers have high aspect ratios, and this increases the wettability of the filler by the matrix, thus creating fewer microvoids and increasing the interaction between the filler and matrix.³⁰ The increased filler–matrix interaction also enables more stress to be transferred from the matrix to the filler during external loading. It can also be seen in Figures 4 and 5 that the stearic acid treatment has a very minimal effect on both the tensile modulus and



Figure 4 Effects of the different filler loadings on the tensile modulus for single-filler and hybrid composites.



Figure 5 Effects of the different filler loadings on the tensile strength for single-filler and hybrid composites.

strength of the composites. The reason for this is that stearic acid acts merely as a nonreactive dispersing agent and not as a coupling agent between the fillers and the matrix. The surfactant molecules are coupled with ionic bonds to the filler surface, and the stearic acid molecules are oriented in directions normal to the surface.¹⁰ As a result of the surface coating, the surface free energy decreases dramatically. A filler treatment with stearic acid reduces particle–particle interactions,⁸ and this leads to better particle dispersion in the PP matrix. However, the introduction of a surfactant also reduces particle–matrix interactions, and this gives rise to increased particle–matrix debonding, thereby reducing the strength of the composite.

The elongation at break drops drastically, especially with increasing talc content, in comparison with unfilled PP, as shown in Figure 6. The decrease in the elongation at break with rigid fillers arises from the fact that the actual elongation experienced by the polymer matrix is much greater than the measured



Figure 6 Effects of the different filler loadings on the elongation at break for single-filler and hybrid composites.



Figure 7 Effects of the different filler loadings on the flexural modulus for single-filler and hybrid composites.

elongation of the specimen. Although the specimen is part filler and part matrix, all the elongation comes from the matrix if the filler is rigid.²⁹ In the case of talc-filled PP composites, there is good adhesion of the fillers to the matrix, the fracture path tends to go from particle to particle, and so a dramatic decrease in deformation can be brought about. CaCO₃ filler particles, however, have poor adhesion to the PP matrix, and so the elongation at break may not decrease as dramatically as that of talc-filled PP composites. Therefore, hybrids with a higher content of CaCO₃ will have better deformability than hybrids with higher concentrations of talc. The incorporation of a filler can also change the mode of failure of PP from ductile behavior to brittle behavior. This reduction in the deformability of PP can be attributed to extensive filler agglomeration, which leads to insufficient homogeneity and also to the rigidity of particulate fillers. Under the influence of an external load, the inhomogeneity of local stress distribution initiates deformation at particular locations in the composite. However, a few compositions with treated CaCO₃ show better ductility than compositions with nontreated CaCO₃. This again shows that the treatment of CaCO₃ fillers with stearic acid reduces particle-matrix interactions and allows more plastic deformation to occur, thereby leading to improved deformability of the composites.

The flexural modulus follows the same trend as the tensile modulus (see Fig. 7): the modulus of the composites increases with increasing talc content. However, the overall flexural modulus values of the composites are lower than the tensile modulus for reasons discussed in earlier studies.¹⁷ Here, the surface treatment of CaCO₃ also shows no significant effect on the modulus of the composite. However, the flexural strength of all the hybrid composites (Fig. 8) is lower

than that of unfilled PP. This could be due to the effects of cavities formed after the incorporation of $CaCO_3$ into the composites. These cavities can be quite large, depending on the size of the $CaCO_3$ fillers. Larger particles contribute to larger cavities. These large cavities can eventually become voids, which have detrimental effects on the composite strength. The increment in the flexural strength of talc-filled PP is also minimal. This can be attributed to the skin formed during the molding of the specimens,²⁹ which is more sensitive to flexural stress. Talc tends to orient with the plane surface parallel to the melt-flow direction during processing at the sample skin;⁹ this causes slippage of the filler against the matrix, thus reducing the reinforcing effect of the filler.

The impact strength has a direct correlation to the filler adhesion to the matrix: a lower filler-matrix adhesion gives better impact resistance.²⁹ Interestingly, the hybrids possess much greater impact strength than unfilled PP and talc-filled PP (see Fig. 9). The drastic decrease in the impact strength of talc-filled PP can be attributed to the immobilization of the macromolecular chains by the filler particles, which limits its ability to adapt to the deformation and, therefore, makes the composite more brittle.¹⁵ The results also suggest that the CaCO₃ filler plays a dominant role in increasing the impact properties of hybrid composites. CaCO₃ has been known to impart toughness to the PP matrix because of the formation of interparticle ligaments, which promote plastic deformation and the transition of the composite from a brittle state to a ductile state.^{10,11} Furthermore, the CaCO₃ particle size is large in light of its isometric shape. Large particles give rise to increased particle-matrix debonding at the interface. As a result of debonding and crazing, large plas-



Figure 8 Effects of the different filler loadings on the flexural strength for single-filler and hybrid composites.

tic deformation can occur in the matrix, being the main energy-absorbing process during impact.

Morphology

Figures 10–12 depict SEM micrographs of T30, UCC30, and TCC30 single-filler composites, respectively. Plastic deformation is very strong in all the fracture surfaces of these specimens studied. The morphologies of UCC30 and TCC30 are almost the same, except that the fillers in TCC30 are perceptibly more homogeneously scattered because of the dispersive effect of stearic acid. The CaCO₃ particles are evidently loosely scattered at the fracture surface, with

large cavities adjacent to the particles. This signifies weak bonding between the filler and matrix, which yields better impact resistance, at the cost of strength, to the composite. The circumstances faced by CaCO₃ composites, however, are contrary to those of T30. Talc fillers are clearly embedded in the polymer matrix, leaving very narrow gaps, if any, between the filler–matrix interfaces. This strong bond between the talc filler and PP matrix, also proven with Pukanszky's quantitative evaluation method, is one of the main reasons for the increase in the composite strength for T30.

Because one of the main objectives of this study was to replace at least 50% of talc with $CaCO_3$, the mor-



Figure 9 Effects of the different filler loadings on the impact strength for single-filler and hybrid composites.



Figure 10 SEM micrograph of 30 wt % talc-filled PP. Filler particles are embedded into the matrix, with very few exposed at the fracture surface, as shown by the arrow.

phologies of the T15UCC15 and T15TCC15 hybrid composites were also studied. Figures 13 and 14 show the micrographs of tensile fracture surfaces for T15UCC15 and T15TCC15, respectively. In both hybrid composites, loose or detached filler particles can hardly be seen. This can be attributed to the nucleating effect of talc, which changes the crystallinity of the polymer, which renders loose or weakly bonded particles to be trapped in the now more rigid polymer network, thus preventing loose particles on the surface even after significant plastic deformation has occurred. The micrographs also clearly demonstrate that the fracture surface of the hybrid composite with stearic acid treated CaCO₃ (T15TCC15) is more brittle and has undergone less plastic deformation than the composite with untreated CaCO₃. From this observation, we can conclude that T15UCC15 has higher impact strength and elongation than T15TCC15. However, the results shown in Figures 6 and 9 are incon-



Figure 12 SEM micrograph of a TCC30 single-filler composite fracture surface.

sistent with this conclusion because both the impact strength and elongation at break of T15TCC15 are almost the same as, if not better than, those of T15UCC15. Again, the particle dispersion and particle-matrix debonding mechanisms play vital roles for both the impact properties and elongation of the composites. As discussed earlier, stearic acid treated CaCO₃ promotes the dispersion of the filler, thereby reducing filler agglomeration; therefore, fewer particles act as stress-concentration points, which are very sensitive to impact. Fillers in hybrids with untreated CaCO₃, though having a higher tendency to agglomerate than those in hybrids with treated CaCO₃, possess almost the same impact strength because of the creation of bigger cavities and higher crazing capabilities, which are also vital to impact-energy absorption.

CONCLUSIONS

Keeping the total filler content of the composites at 30 wt %, while talc was partially replaced with CaCO₃, has given a true picture of how the different fillers



Figure 11 SEM micrograph of 30 wt % CaCO₃-filled PP. The bonding of the filler to the matrix is very weak because of the formation of cavities around the filler particles, as shown by the arrows.



Figure 13 SEM micrograph of a T15UCC15 hybrid composite fracture surface.



Figure 14 SEM micrograph of a T15TCC15 hybrid composite fracture surface.

interact with each other and with the PP matrix. Most of the hybrid composites successfully maintained certain properties; this depended on the type of filler that was more dominant and the influence of the individual filler itself on the properties of PP. Basically, talcdominant hybrid composites had higher tensile and flexural strength and modulus, whereas CaCO₃-dominant hybrids were tougher and more deformable. A hybridization effect was seen when 50% of talc was replaced with CaCO₃, as there was a more significant increase in the flexural and impact strength than in other hybrid compositions. Although the incorporation of CaCO₃ through the hybridization process tends to reduce the modulus and tensile and flexural strength, its role in improving the brittleness problem faced by typical talc-filled PP composites is inevitable. Both talc and CaCO₃ fillers also had significant nucleating effects on the PP matrix, which contributed to the increase in modulus and the retention of the strength of the composites.

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